

(12) **UK Patent Application** (19) **GB** (11) **2 263 856** (13) **A**
(43) Date of A publication 11.08.1993

(21) Application No 9202195.5

(22) Date of filing 01.02.1992

(71) Applicant
Pauls Malt Limited

(Incorporated in the United Kingdom)

**P O Box 39, 47 Key Street, Ipswich, Suffolk, IP4 1BX,
United Kingdom**

(72) Inventor
Ian Charles Cantrell

(74) Agent and/or Address for Service
Dummett Copp & Co
**25 The Square, Martlesham Heath, Ipswich, IP5 7SL,
United Kingdom**

(51) INT CL⁵
A23L 1/185 1/10

(52) UK CL (Edition L)
A2B BMMA

(56) Documents cited
GB 0311424 A

(58) Field of search
**UK CL (Edition L) A2B BMMA BMR1 BMRS BMW
BMX
INT CL⁵ A23L**

(54) **A foodstuffs additive**

(57) A foodstuffs additive which has a high reducing power is prepared from a mash of roasted barley and/or malt by a process which comprises the steps of preparing a mash, screening it to separate a liquid from the insoluble matter 18, and subjecting the resulting liquid to a cross-flow membrane filtration process. This process separates the liquid into a first fraction 22 which is high in reducing power, and a second fraction 24.

At least one drawing originally filed was informal and the print reproduced here is taken from a later filed formal copy.

The claims were filed later than the filing date within the period prescribed by Rule 25(1) of the Patents Rules 1990.

GB 2 263 856 A

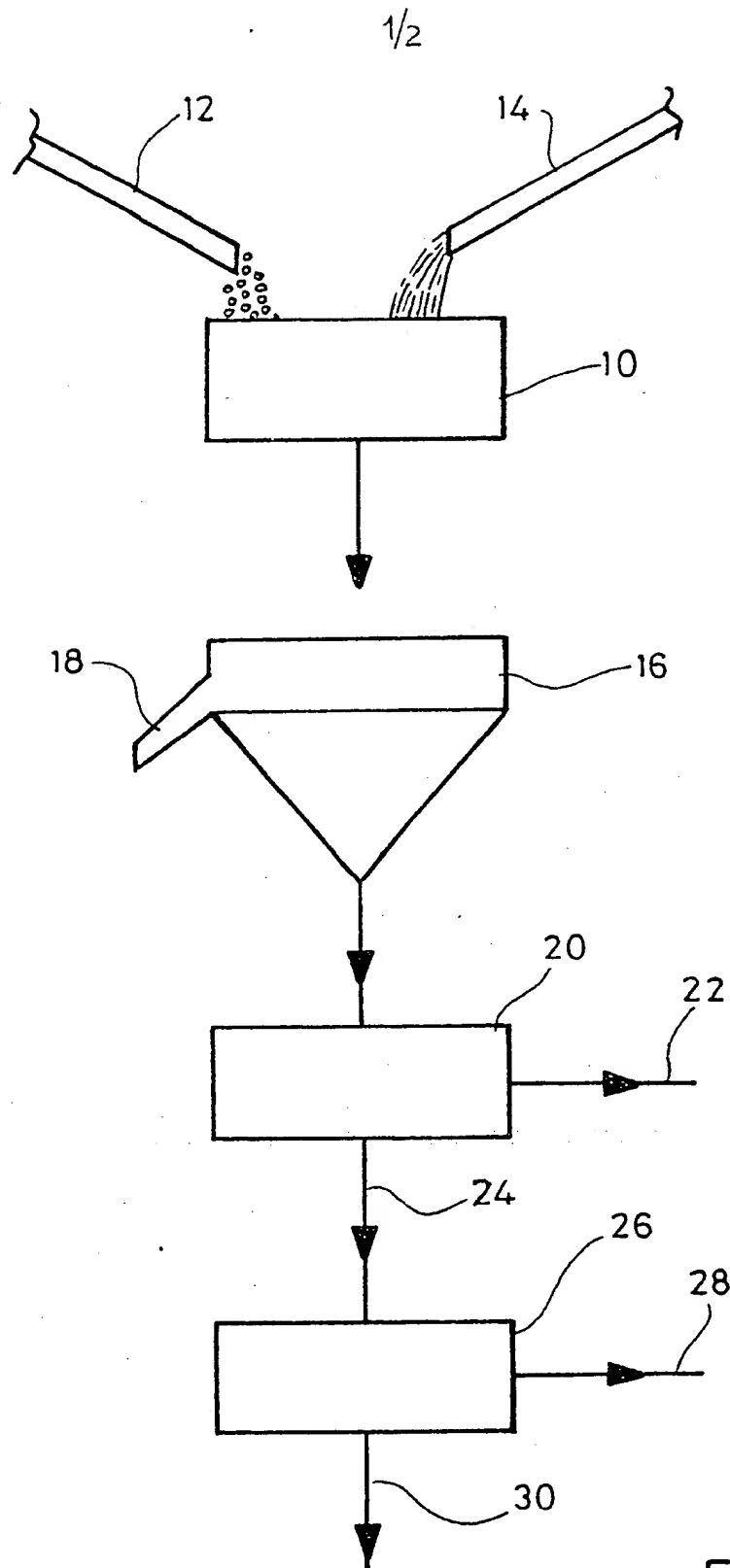


Fig. 1

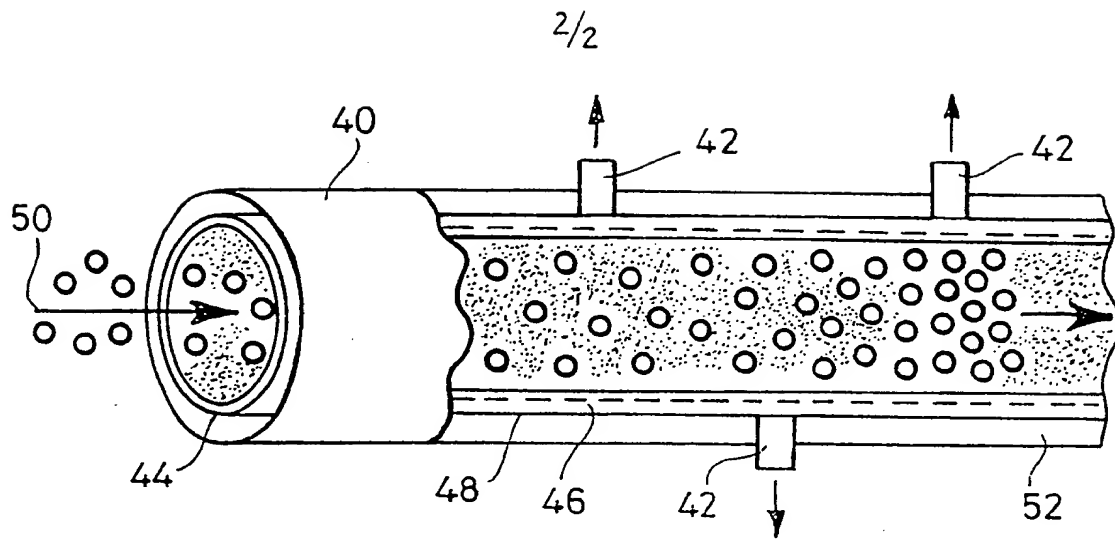


Fig. 2

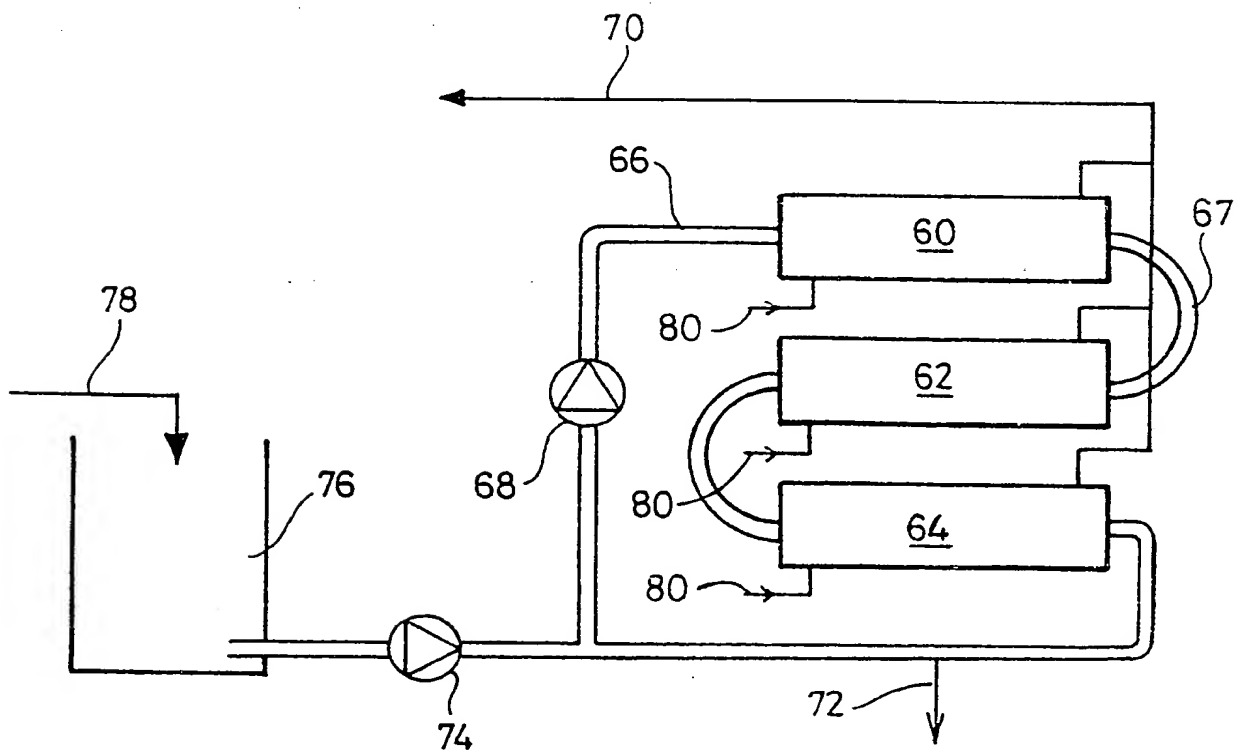


Fig. 3

A FOODSTUFFS ADDITIVE

- This invention relates to a foodstuffs additive which has a high reducing power. The additive will be added to foodstuffs, particularly but not exclusively in the brewing industry, to delay oxidation of foodstuffs. The invention also provides a process for preparing the additive, and extend to the additive as an anti-oxidant.
- 10 It has long been known as desirable to add anti-oxidants to beers and other foodstuffs in order to delay the onset of oxidation so that the beer can be kept for longer periods without deteriorating. Known anti-oxidants (eg ascorbic acid and sodium sulphite) are synthetically produced
- 15 chemicals. In all areas of foodstuffs production and particularly in beer making there is a desire to avoid additives in the form of synthetic materials, and to use only naturally occurring materials.
- 20 It is therefore an object of this invention to extract from naturally occurring substances a substance which is high in reducing power, and which can therefore be used as an anti-oxidant.
- 25 According to the present invention, there is provided a process for producing a foodstuffs additive which is high in reducing power, the process comprising the steps of preparing a mash of either roast barley or malt or of both roast barley and malt, screening the mash to separate a
- 30 liquid from the insoluble matter in the mash, subjecting the resulting liquid to a cross-flow membrane filtration process to separate the liquid into a first fraction high in reducing power and a second fraction.

The malt may be roast malt which has been roasted before being mashed.

The first fraction will be high in colour in addition to being high in reducing power. This may be acceptable in some foodstuffs applications, but may not be acceptable in other applications, eg brewing where a highly coloured additive would adversely affect the final product.

The second fraction, although being low in absolute reducing power in comparison with the first fraction, has a ratio of reducing power to colour which is higher than that of the first fraction. The second fraction is also relatively high in flavour. It is possible, within the scope of the invention, to further treat the second fraction to reduce its flavour so as to produce an additive which is high in reducing power relative to its flavour and colour and which is therefore useful in foodstuffs where neither flavour nor colour should be added with the additive.

Preferably the cross-flow membrane filtration process is an ultrafiltration process. The ultrafiltration membrane may have a molecular cut-off of 20,000 or less but preferably has a molecular cut-off of 10,000 or less. Particularly satisfactory results have been achieved with ultrafiltration membranes having a molecular cut-off of 6,000 molecular weight.

Treatment of the second fraction to reduce flavour is preferably carried out by a reverse osmosis process, making use of a membrane. The membrane for the reverse osmosis process preferably has a molecular cut-off below 500, and a molecular cut-off of 200 is particularly suitable. Both the ultrafiltration and the reverse osmosis processes are carried out using a re-circulation circuit from which

permeate and concentrate are continuously withdrawn, and to which feed is continually added to maintain a constant volume in circulation.

- 5 The invention also extends to foodstuffs additives prepared by the processes set forth above. The additives may be in the form of a liquid, or may be further processed to produce a solid, particulate form, eg by freeze drying.
- 10 The invention also extends to the use of an extract produced from a mash of malt and/or roast barley as an anti-oxidant for foodstuffs.

The invention will now be further described, by way of
15 example, with reference to the accompanying drawings, in which:

Figure 1 is a schematic flow diagram illustrating the preparation process in accordance with the invention;

20

Figure 2 shows a typical device for carrying out ultrafiltration or reverse osmosis via a membrane; and

25

Figure 3 shows a re-circulation circuit including filters of the type illustrated in Figure 2.

30

Figure 1 shows a mash tun 10 which is fed with malt or roast barley through an inlet 12 and with water through a pipe 14. The water may be hot or cold. Following a conventional mashing process, the mash is discharged onto a rough screen filter 12 so that the solids can be separated from the liquid. The solids are retained by the filter and are then diverted to waste through an outlet 18.

The crude malt extract in the form of a liquid including fine particulate passes into an ultrafiltration unit 20 where a separation takes place with the fine particulate and a fraction of high molecular weight being collected on one side of the filter and then leaving the unit as a retentate 22. A permeate 24 passes through the filter and consists of a liquid containing only relatively low molecular weight components. The permeate 24 passes into a reverse osmosis unit 26 where a further separation takes place via a filter membrane which is finer than the membrane used in the ultrafiltration unit 20. The retentate 28 and the permeate 39 from this reverse osmosis step differ in the molecular weight of the molecules held in solution.

It is found that if the ultrafiltration membrane in the ultrafiltration unit 20 has a rating of 6,000 molecular weight, then the permeate 24 is high in malt flavour, whereas the retentate 22 is high in colour. Tests have also shown that the high colour, retentate fraction is high in reducing power and is therefore useful as an anti-oxidant.

Furthermore, when the permeate 24 is subjected to a reverse osmosis process in the unit 26, then the permeate 30 has a significantly higher ratio of reducing power to colour and to flavour than the retentate 28.

Ultimately it is desirable to produce a fraction from naturally occurring malt which is high in reducing power but low in colour and low in flavour. Such an additive can be added to foodstuffs without affecting the colour or the flavour of the resulting foodstuffs product.

Both the units 20 and 26 conveniently take the form of cross-flow filtration units which will be described in more detail with reference to Figures 2 and 3. Figure 2 shows a

filtration unit which has an outer tube 40 with radial outlet passages 42 for permeate. Within the tube 40 is an inner tube 44, the walls of which are formed by a membrane 46 supported by a membrane carrier 48. The fluid to be
5 filtered passes through the inner tube 44 in the direction indicated by the arrow 50. Pressure is applied in the annular space 52 between the inner 40 and outer 44 tubes, so that there is a pressure differential across the membrane 46. As a result, liquid passes through the membrane 46 and
10 exits through the outlets 42.

The openings in the membrane 46 are however very small. Where the membrane has, for example, a rating of 6,000 molecular weight, then only molecules with a molecular
15 weight below this figure can pass through the membrane and all solids and molecules of higher molecular weight are retained within the inner tube 44 and are continuously circulated through the system. Those skilled in the art will however be aware that the size of the molecules which
20 will pass through or will be retained by the membrane are only approximately related to their molecular weight. Membrane specifications or membrane pore sizes are however conventionally given in terms of molecular weight. As re-circulation proceeds, the concentration of fine solids and
25 high molecular weight molecules in the feed increases, and a concentrate can be removed from the system as retentate.

Figure 3 shows a circuit diagram where three filtration units 60, 62, 64 are mounted in series and are connected in
30 a circuit 66 by a feed circulation pipe 67. A circulation pump 68 pumps fluid around the circuit, and permeate is taken off from each unit 60, 62, 64 and passed to a common outlet pipe 70. The circuit 66 also has an outlet 72 for concentrate. The volume of fluid in the circuit 66 is

maintained by a feed pump 74 which draws fluid from a holding tank 76 which in turn is fed by a fluid inlet 78.

5 The filtration units 60, 62, 64 are each supplied with pressure through supply pipes indicated schematically at 80. The filter membranes in the three units 60, 62, 64 will all be of the same rating, ie of the same pore size. Where two graded filtration steps take place, the permeate from the first stage (eg ultrafiltration) can be fed to a second
10 filtration stage (eg reverse osmosis) with finer filtration membranes.

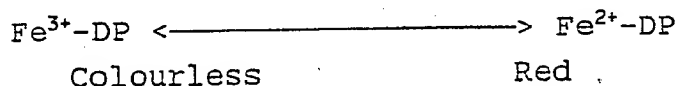
In an example of the process according to the invention, the mashing process in the tun 10 was carried out at a
15 temperature of 30°C. The rating of the ultrafiltration membrane in the ultrafiltration unit 20 was 6,000 molecular weight, and the rating of the reverse osmosis membrane in the unit 26 was 200 molecular weight.

20 Within the filtering stage, suitable results were obtained at temperatures of between 20° and 50°C and at pressures of 35-55 bar. The fluid circulation speed within the circuit 66 is typically of the order of 2.5-5 metres per second. A typical maximum permeate flux for this system is in the
25 order of 10 l/m²/h.

The product, ie the retentate 22 from the ultrafiltration and/or the permeate 30 from the reverse osmosis stage consistently had a significantly improved reducing power.
30 For the purposes of these tests, reducing power was determined by the method described by Chapon, L., Louis, C and Chapon, S (1971) Proceedings of the European Brewery Convention Congress, Estoril, 307-322.

In this method the presence of reducing compounds results in the reduction of Fe^{3+} - α, α' -dipyridyl(Fe^{3+} -DP) from the oxidised, colourless form to the reduced, red-coloured, Fe^{2+} form.

5



10 The absorbance of the reduced solution is determined spectrophotometrically at 510 nm after a 3 minute incubation period and the absorbance x 1000 taken as a measure of relative reducing power.

15 By way of example, in one experiment using this method, the reducing power of the raw material (in this case roast barley) was 6.3 units per gram. The reducing power of the retentate 22 was 570 units per gram and the reducing power of the permeate 30 was 190 units per gram.

20 The product as produced direct from the filtration stages is in the form of a liquid. However this liquid can be converted into particulate form, for example by freeze drying to produce the additive in a form in which it can be easily used by foodstuffs manufacturers, particularly
25 brewers.

It is a particular advantage of this product that it is produced entirely from natural material and indeed is a by-product of the malt which is used in any case in brewing.
30 The ultrafiltration and reverse osmosis steps are useful in separating the reducing power fraction from, respectively, the flavour components of the malt and the colour components of the malt.

35

CLAIMS

1 A process for producing a foodstuffs additive which is
high in reducing power, the process comprising the steps of
5 preparing a mash of either roast barley or malt or of both
roast barley and malt, screening the mash to separate a
liquid from the insoluble matter in the mash, subjecting the
resulting liquid to a cross-flow membrane filtration process
to separate the liquid into a first fraction high in
10 reducing power and a second fraction.

2 A process according to claim 1 wherein the malt has
been roasted before being mashed.

15 3 A process according to claim 1 or claim 2, wherein the
cross-flow membrane filtration process is an ultrafiltration
process.

4 A process according to claim 3 wherein the
20 ultrafiltration filter has a molecular weight cut-off of
20,000 or less.

5 A process according to claim 3 wherein the
ultrafiltration filter has a molecular weight cut-off of
25 10,000 or less.

6 A process according to claim 3 wherein the
ultrafiltration filter has a cut-off of 6,000 molecular
weight.

30

7 A process according to any of the preceeding claims,
wherein the second fraction is further treated to reduce
flavour by a reverse osmosis process, making use of a
membrane.

35

8 A process according to claim 7 wherein the membrane for the reverse osmosis process has a molecular weight cut-off below 500.

5 9 A process according to claim 7 wherein the membrane for the reverse osmosis process has a molecular weight cut-off of 200.

10 10 A process according to any of the preceeding claims, wherein the separation processes are carried out using a recirculation circuit from which permeate and concentrate are continuously withdrawn, and to which feed is continually added to maintain a constant volume in circulation.

15 11 A foodstuffs additive prepared according to any of the preceeding claims.

12 12 A solid foodstuffs additive obtained by drying a liquid additive prepared according to any of the preceeding claims.

20 13 An extract produced from a mash of malt and/or roasted barley for use as an anti-oxidant for foodstuffs.

14 14 A process substantially as herein described with
25 reference to the accompanying drawings.

Examiner's report to the Comptroller under
Section 17 (The Search Report)

Application number

GB 9202195.5

Relevant Technical fields

(i) UK Cl (Edition L) A2B: BMMA; BMR1; BMR5; BMX;
BMW

(ii) Int Cl (Edition 5) A23L

Search Examiner

B J GARDNER

Date of Search

29 APRIL 1993

Databases (see over)

(i) UK Patent Office

(ii) NONE

Documents considered relevant following a search in respect of claims 1 TO 14

Category (see over)	Identity of document and relevant passages	Relevant to claim(s)
A	GB 0311424 (LAHMANN AND LAHMANN) see whole document	1 at least

Category	Identity of document and relevant passages	Relevant to claim(s)

Categories of documents

X: Document indicating lack of novelty or of inventive step.

Y: Document indicating lack of inventive step if combined with one or more other documents of the same category.

A: Document indicating technological background and/or state of the art.

P: Document published on or after the declared priority date but before the filing date of the present application.

E: Patent document published on or after, but with priority date earlier than, the filing date of the present application.

&: Member of the same patent family, corresponding document.

Databases: The UK Patent Office database comprises classified collections of GB, EP, WO and US patent specifications as outlined periodically in the Official Journal (Patents). The on-line databases considered for search are also listed periodically in the Official Journal (Patents).